Universidade de Lisboa – Faculdade de Medicina Dentária

Influence of exposure time and light intensity on depth of cure of Bulk-fill resin composite

Frederico de Almeida Portugal Catalão

Dissertação

Mestrado Integrado em Medicina Dentária

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Influence of exposure time and light intensity on depth of cure of Bulk-fill resin composite

Dissertação orientada pelo Prof. Doutor Jaime Pereira Fontes de Almeida Portugal

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Aos meus pais,

À Mafalda, José e Salvador, meus irmãos,

À Raquel, com quem vou casar este ano.

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Resumo:

As restaurações de dentes posteriores com resina composta tornaram-se uma prática clínica diária em Medicina Dentária, devido à crescente exigência estética dos pacientes. As características mecânicas das resinas compostas atualmente disponíveis no mercado têm permitido a sua utilização com um bom prognóstico a médio e longo prazo**.** Devido às suas caraterísticas, a maioria dos compósitos utilizados para estes fins tem uma reação de polimerização ativada por luz. Sendo conhecida a diminuição da intensidade da radiação luminosa à medida que atravessa a resina composta, este material apresenta tradicionalmente uma profundidade de polimerização de 2 mm. Tal facto, aliado à contração de polimerização, condiciona a sua forma de aplicação clinica, obrigando à sua aplicação por incrementos. A utilização desta técnica conduz a um aumento do tempo despendido na realização dos procedimentos clínicos, com todos os efeitos adversos que daí poderão surgir. Outra desvantagem atribuída à técnica incremental é a maior susceptibilidade à contaminação e introdução de poros. Recentemente, têm surgido no mercado alguns compósitos para os quais os respetivos fabricantes reclamam uma reduzida contração e uma elevada profundidade de polimerização. Algumas destas resinas compostas, apelidadas de *bulk-fill*, foram desenvolvidas como material de restauração único (*fill*). Outras (*base*), devem ser utilizados para preenchimento da cavidade a restaurar, mas devem ser revestidos por uma resina composta tradicional, que apresente uma maior resistência ao desgaste. As resinas compostas *fill* parecem apresentar maior quantidade de partículas de carga, o que lhes confere maior resistência à abrasão e maior valor de microdureza. Segundo os respetivos fabricantes, estas resinas compostas poderão ser corretamente fotopolimerizados em incrementos de 4 ou 5 mm de espessura. Desta forma será possível a redução dos incrementos necessários, o que torna as consultas mais curtas e mais confortáveis para os pacientes. Uma polimerização adequada resulta em melhores propriedades físicas e adaptação marginal. Por outro lado, uma polimerização inadequada pode resultar em insuficiente conversão dos componentes da matriz orgânica da resina composta, resultando num aumento da quantidade de monómero residual. As propriedades mecânicas das resinas compostas dependem não só das partículas de carga que compõem a sua parte inorgânica, mas também de algumas características da luz (intensidade, comprimento de onda e tempo de exposição), e do grau de conversão. Tem

sido demonstrada uma correlação positiva entre a microdureza e o grau de conversão das resinas compostas. A eficácia na polimerização ao longo de um incremento pode ser medida direta ou indiretamente. Os métodos diretos não são utilizados tão frequentemente, devido à sua complexidade, custo e tempo que consomem. Os métodos indiretos, como o cálculo do rácio de microdureza e o método de raspagem, por exemplo, são frequentemente utilizados. Apesar de o método de raspagem ser o indicado pela ISO, o rácio de microdureza tem sido o método indireto preferido por grande parte dos investigadores, por ter ser menos permissivo e por ter sido demonstrada a existência de uma correlação entre este método e a espetroscopia de infra-vermelhos, para a determinação da profundidade de polimerização. Para o cálculo do rácio de microdureza deverá ser determinada a microdureza da superfície mais perto da fonte luminosa utilizada como ativador da reação de polimerização e a microdureza do compósito à profundidade pretendida. Para se considerar que o incremento de compósito se encontra polimerizado de forma adequada, o rácio de microdureza deverá ser superior ou igual a 0,80. Os estudos mais recentes acerca destas resinas compostas *bulk-fill*, além de serem escassos, apresentam resultados controversos. Assim, são necessários estudos adicionais, para compreender se de facto é possível atingir elevada profundidade de polimerização sob as condições recomendadas pelos fabricantes.

Objectivos: Avaliar influência do método de fotopolimerização na microdureza e na profundidade de polimerização de seis resinas compostas *bulk-fill*. Comparar dois métodos utilizados para determinar a profundidade de polimerização das resinas compostas.

Materiais e métodos: A profundidade de polimerização dos compósitos foi determinada através do método recomendado pela ISO 4049 e pela determinação do rácio de microdureza. As resinas compostas utilizadas foram: Tetric EvoCeram Bulk Fill (Ivoclar Vivadent), x-tra base (Voco), x-tra fill (Voco), Filtek Bulk Fill (3M ESPE), SonicFill (Kerr), SDR (Dentsply). Para cada uma destas resinas compostas, foram criados 4 grupos experimentais, consoante o método de polimerização utilizado (600mW/cm^2 durante 20 s; 1200mW/cm^2 durante 10 s; 600mW/cm^2 durante 40 s; 1200mW/cm^2 durante 20 s). Para a determinação da microdureza da superfície de compósito mais próxima da fonte de luz (topo) e para o cálculo do rácio da microdureza foram preparados 5 espécimes por grupo experimental, com 2x2x7 mm, utilizando um molde de teflon (n=5). Imediatamente após a irradiação luminosa, o valor de microdureza

Knoop foi determinado, com um microdurometro (Duramin Struers A/S, DK-2750 Ballerup, Denmark), realizando três indentações a cada profundidade (no topo, e na superfície lateral a 1, 2, 4 e 5 mm de profundidade), com uma carga de 98,12 mN durante 10 s. Para cada profundidade, foi calculado o rácio relativamente ao topo do espécime. Um rácio de microdureza superior a 0,80 foi considerado indicativo de correta polimerização a essa profundidade. Para a determinação da profundidade de polimerização pelo método recomendado pela norma ISO 4049, foram preparados 2 espécimes por cada grupo experimental (n=2), utilizando um molde de aço. Os espécimes cilíndricos com dimensão padronizada (4 mm de diâmetro e 10 mm ou 12 mm de altura, conforme a profundidade de polimerização da resina composta fosse de 4 ou 5mm, respectivamente) foram fotopolimerizados aplicando uma fonte luminosa numa das extremidades. Após a remoção do molde de aço, todo o compósito não endurecido foi descartado com a ajuda de uma espátula de plástico rígido. Com a ajuda de um micrótomo digital foi calculada a altura do cilindro de compósito remanescente. Metade dessa altura foi considerada a profundidade de polimerização. Os dados de microdureza obtidos foram analisados com recurso a testes não paramétricos Kruskal-Wallis seguido de comparações múltiplas de acordo com o método LSD às ordens.

Resultados: Os valores de microdureza do topo variaram entre 14,2 KHN para a resina composta Filtek^{TM} Bulk Fill fotopolimerizado durante 10 segundos com uma intensidade de 1200 mW/cm^2, e 51,4 KHN para a resina composta SonicFill^{TM}, com 1200 mW/cm^2 durante 20 segundos. A análise dos resultado com o método de Kruskal-Wallis revelou uma influência estatisticamente significativa ($p < 0,001$) do compósito sobre os valores de microdureza obtidos no topo dos espécimes. As resinas compostas indicadas para utilização como base (x-tra base, Filtek Bulk Fill e SDR) apresentaram valores de microdureza estatisticamente mais baixos ($p < 0,05$) que as concebidas para serem utilizadas sem uma resina composta tradicional como revestimento (x-tra fil, Tetric EvoCeram Bulk Fill e SonicFill). Não foram observadas diferenças estatisticamente significativas entre os diferentes métodos de polimerização ($p = 0,244$). A profundidade de polimerização segundo o método recomendado pela norma ISO variou entre os 2,92 mm para o Tetric Evoceram Bulk Fill fotopolimerizado com 600 mW/cm^2 durante 20 segundos, e os 4,97 mm para o x-tra base com 600mW/cm^2 durante 40 segundos. A profundidade de polimerização obtida com o rácio de microdureza variou entre 4 mm, para o x-tra fil (1200 mW/cm^2 – 20 s) e o Filtek Bulk Fill (600

mW/cm^2 – 20 s; 600 mW/cm^2 – 40 s; 1200 mW/cm^2 – 20 s), e 1 mm para o Tetric Evoceram Bulk Fill (600 mW/cm^2 – 20 s; 1200 mW/cm^2 – 10 s).

Conclusões: As resinas compostas utilizadas como *fill* apresentam uma microdureza superior às utilizadas como *base*. A microdureza das resinas compostas não foi afetada pelo método de polimerização. Nem todos os compósitos apresentam a profundidade de polimerização reclamada pelos fabricantes. O método recomendado pela norma ISO 4049 permitiu obter uma maior profundidade de polimerização superior ao obtido pelo rácio de microdureza.

Abstract:

Objectives: To evaluate influence of the light curing method on the microhardness and the depth of cure of six bulk-fill composite resins. To compare two methods used to determine the depth of cure of composite resins.

Materials and Methods: Depth of cure was determined according to ISO-4049 and microhardness ratio. Specimens were divided by 24 experimental groups according to the possible combinations between composite [Tetric EvoCeram Bulk Fill (Ivoclar-Vivadent), x-tra base (Voco), x-tra fil (Voco), Filtek Bulk Fill (3M-ESPE), SonicFill (Kerr) and SDR (Dentsply) and curing method (600mW/cm^2–20s; 1200mW/cm^2–10s; 600mW/cm^2–40s; 1200mW/cm^2–20s)]. Using a teflon mould, specimens (2x2x7 mm) were made to analyse Knoop microhardness on the top of the specimens and depth of cure according to microhardness ratio (n=5). Two specimens per group were made to determine depth of cure by ISO-4049 instructions (n=2). Microhardness data were analysed with Kruskal-Wallis followed by LSD post-hoc tests ($p<0.05$).

Results: Microhardness values ranged from 14.2 KHN (Filtek Bulk Fill, 1200mW/cm^2–10s) to 51.5 KHN (SonicFill, 1200mW/cm^2–20s). Base composite resins (x-tra base, Filtek Bulk Fill e SDR) showed a statistically ($p<0.05$) lower microhardness then fill composites (x-tra fil, Tetric EvoCeram Bulk Fill and SonicFill). No significantly differences were found between the curing methods ($p=0.244$). ISO depth of cure ranged between 2.92 mm (Tetric Evoceram Bulk Fill – 600mW/cm^2–20s) and 4.97 mm (x-tra base – 600mW/cm^2–40s). Ratio depth of cure ranged from 4 mm [x-tra fill - 1200mW/cm^2-20s, Filtek Bulk Fill (600mW/cm^2-20s; 600mW/cm^2-40s; 1200mW/cm^2-20s)] to 1 mm [Tetric Evoceram Bulk Fill – (600mW/cm^2-20s; 1200mW/cm^2-10s)].

Conclusion: Fill composite resins microhardness was higher than base composites. The top microhardness was not affected by the curing method. Not all composites yielded the depth of cure claimed by manufactures. ISO 4049 recommended method afforded higher depth of cure than the one obtained by microhardness ratio tests.

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Introduction:

Restore teeth with composite resin have become daily procedures for most dentists, mainly due to a remarkable development of adhesive systems, more aesthetic requirements of patients, and the concern to preserve as much tooth structure as possible. The mechanical properties of composites, such as abrasion resistance, have increased since they started being used as restorative materials, becoming usable to restore posterior teeth with good long term prognostic (Arikawa *et al*, 2011; Alpöz *et al*, 2008; Camargo *et al*, 2009; El-Safty, Silikas and Watts, 2012; Hedge, Hedge and Malhan, 2008; Kwon, Ferracane and Lee, 2012; Leprince *et al*, 2012; Poggio *et al*, 2012; Yaman *et al*, 2011).

Due to its characteristics, the polymerization of most composite resins used for these purposes is activated by light. As known, the intensity of light radiation decreases as it penetrates through the composite. So, this material traditionally has a 2 mm depth of cure. This fact, coupled with the polymerization shrinkage, conditions its clinical application to be incremental. The incremental technique involves placing increments of composite resin with maximum thickness of 2 mm, followed by curing from an occlusal direction, repeatedly.

The use of this technique leads to an increase in time spent with clinical procedures, with all the adverse effects that may arise therefrom. Other disadvantages of this technique are the introduction of pores or contamination between layers, failures in adhesion between layers, difficulty in placement of increments in cavities of small dimensions, and the time consumed (El-Safty, Silikas and Watts, 2012; Rouhollahi, Mohammadibasir and Talim, 2012). Recently, some composite resins have appeared, for which the respective manufacturers claim reduced shrinkage and high depth of cure. According to the respective manufacturers, these composite resins “bulk-fill” may be properly photoactivated in increments of 4 or 5 mm thickness. This will allow the reduction of increments, which makes appointments shorter and more comfortable for patients. Some of these are used as the only restorative material in the cavity (fill), and others require a conventional coating composite (base).

Tetric EvoCeram® Bulk Fill (Ivoclar Vivadent®) is a nano-hibrid resin composite, adapted to bulk filling technique. It contains a polymerization booster

(Ivocerin) that allows that layers with a thickness of 4 mm can be cured in 10 seconds. There are three shades available: IVA, IVB e IVW. For a light intensity of 500 mW/cm^2 or higher, 20 seconds irradiation is recommended. For a light intensity of 1000 mW/cm^2 or higher, 10 seconds irradiation is recommended.

x-tra base® (Voco®) is a flowable resin composite, to be used as base. There are two shades available: Universal and A2. It is possible to achieve the complete polymerization of a 4 mm thickness increment in 10 seconds, using any light intensity, for the shade U. For the shade A2, with a light intensity between 500 and 800 mW/cm^2, an irradiation of 40 seconds is recommended. For a light intensity higher than 800 mW/cm^2 a 20 seconds irradiation is recommended.

x-tra fil (Voco®) is a light curing resin composite, indicated to Class I and II posterior teeth restorations. In 10 seconds it is possible to achieve the complete polymerization of a 4 mm thickness increment. For a light intensity higher than 800 mW/cm^2 a 10 seconds irradiation is recommended, and with a light intensity between 500 and 800 mW/cm^2, an irradiation of 20 seconds is recommended.

Filtek™ Bulk Fill (3M ESPE) is a flowable resin composite, than can be applied in increments with a thickness of 4 mm, at the bottom of the cavities. There are 4 available shades: Universal, A1, A2 e A3. For a light intensity of 1000 mW/cm^2 the curing time should be 10 seconds for shade U and 20 seconds for shades A1, A2 e A3. For a light intensity of 550 mW/cm^2 the curing time should be 20 seconds for shade U and 40 seconds for shades A1, A2 e A3.

SonicFill™ (Kerr™) is a nano-hibrid resin Bulk-fill composite that can be applied in a single increment with a thickness of 5 mm, achieving complete polymerization after 20 seconds light exposure. This material is available in 4 shades: A1, A2, A3 and B1. For a light intensity higher than 550 mW/cm^2, 20 seconds of irradiation are enough for any of the four shades.

SDR™ (Dentsply) is a flowable resin composite, to be used as base, and can be applied in a single increment with a thickness of 4 mm, achieving complete polymerization after 20 seconds light exposure, with a light intensity higher than 550 mW/cm^2. Only Universal shade is available.

Recent studies with various types of bulkfill composite resins, that compared them with the conventional incremental technique composite resins, have evaluated the polymerization stress, hardness, microleakage, marginal adaptation and interfacial tension. However, as many studies have been performed using hybrid composites and modifying only the technique, the bulk-fill composite resins themselves lack studies (El-Safty, Silikas and Watts, 2012).

Garoushi *et al* (2013) measured the depth of cure of x-tra base, SDR, Filtek Bulk Fill, Tetric EvoCeram Bulk Fill and SonicFill by ISO and found that x-tra base, SDR, Filtek Bulk Fill achieved the value stated by manufacturer, but the others didn't. Ilie *et al*, 2013 studied the depth of cure of Tetric EvoCeram Bulk Fill and x-tra base, and found that both enable at least 4 mm. Czasch and Ilie, 2013 studied other Bulk Fill resin based composites and found that these materials enabled a depth of cure of 4 mm.

Studies with bulk-fill composite resins are still few, and their results controversial, which justifies the need for further studies with these materials.

The physical properties of composite resins are dependent on the degree of conversion of the resin matrix. A positive correlation between hardness and degree of conversion has been demonstrated.

The effectiveness in the polymerization along the composite resin can be measured directly or indirectly. Direct methods are not routinely used, due to their complexity, cost and time consumed. Indirect methods, scraping or microhardness ration, for example, are commonly used. The microhardness is a good indicator of the depth of cure of the light-curing composite resins and there is a good correlation between hardness ratio and infrared spectroscopy (Flury *et al*, 2012; Hedge, Hedge and Malhan, 2008; Lima *et al*, 2012; Poggio *et al*, 2012; Yaman *et al*, 2011).

Although scraping method is indicated by ISO, microhardness ratio has been used as the preferred indirect method by most researchers , for being less permissive and for its correlation with infra-red spectroscopy for determination of depth of cure. To calculate the microhardness ratio, the microhardness of the surface closer to the light source used as an activator of the reaction and the microhardness of the composite to the desired depth must be determined. To define the depth of cure based on top and bottom

hardness measurements, it is common to calculate the ratio of bottom/top hardness. To consider that the bottom is sufficiently cured, ratio values should be higher than 0.80 (Borges *et al*, 2009; Flury *et al*, 2012; Heintze and Zimmerli, 2011; Leprince *et al*, 2012; Moore *et al*, 2012; Poggio *et al*, 2012).

"ISO 4049; Depth of cure" method is used to define the maximum thickness of each increment. The resin composite to be tested is inserted into a tubular mould, cured and removed from the mould. Thereafter, the uncured material is removed with a spatula and leaving a rigid cylindrical specimen. The final length of the specimen is measured and divided into two. The value found is taken as the depth of cure and sets the maximum thickness of each increment (Flury *et al*, 2012; Leprince *et al*, 2012; Lima *et al*, 2012; Moore *et al*, 2012). It is still necessary to understand if this method is suitable for composite resins used nowadays, such as Bulk-fill (Flury *et al*, 2012; Lima *et al*, 2012).

A study of Flury et al, 2012, was intended to understand if, for Bulk-fill composites, the depth of cure determined by ISO 4049 method accurately reflected in the results of depth of cure obtained from Vickers method, concluding that ISO 4049 method overestimates the depth of cure when compared to determining the estimates of Vickers hardness by the degree of conversion (Lima *et al*, 2012).

Recent studies report that introduction of fewer increments, or even bulk filling may have identical results, since the composite resin is prepared to be used with the chosen technique. (El-Safty, Silikas and Watts, 2012). Some manufacturers have developed "bulk-fill" composite resins that can be applied in layers in Class I and II cavities, 4 mm thick, with an adequate degree of conversion and controlled tension. This technique makes appointments shorter and more comfortable for patients. Many authors consider that "bulk-fill" composite resin must have some important features such as low polymerization shrinkage, less viscosity so that they can be adapted to the cavity, be easily dispensable, easy to handle, have good physical properties and depth of cure of at least 4 mm. (Czasch and Ilie 2012; El-Safty, Silikas and Watts, 2012; Flury *et al*, 2012).

Some of these composite resins, called "bulk-fill", were developed as single restoring material (fill). Others must be used for cavity filling but must be coated with a traditional composite (base).

Fill composites appear to have a greater percentage of filler particles than base composites, giving them greater abrasion resistance and top microhardness (Garoushi *et al*, 2013).

An adequate cure results in better physical properties and marginal adaptation. Inadequate curing can result in insufficient conversion of the components of the resin, which results in an increased amount of residual monomer (Arikawa *et al*, 2011; Camargo *et al*, 2009; El-Safty, Silikas and Watts, 2012; Rouhollahi, Mohammadibasir and Talim, 2012; Yaman *et al*, 2011; Moore *et al*, 2008). The mechanical properties of composite resins depend not only on the charge particles that compose the inorganic part, but also on some light characteristics (intensity, wavelength and exposure time) (El-Safty, Silikas and Watts, 2012; Kuguimiya *et al*, 2010).

The intensity of the light emitted by the light curing units depends on several factors, and the total amount of energy influences mechanical properties of composite resins. The distance between the light curing unit and the resin composite is also extremely important (Leprince *et al*, 2012; Lima *et al*, 2012; Poggio *et al*, 2012). The technology in this area has evolved considerably over the years, with the appearance of high intensity halogen lights, plasma arc lights and light emitting diode units (LEDs) (Mousavinasab and Meyers, 2011; Poggio *et al*, 2012). The halogen lights are the most used and the most economic, but its irradiance decreases due to bulb and filter ageing (Voltarelli *et al*, 2009). The power of blue LEDs falls within the absorption spectrum of camphorquinone iniciator (400-500 nm), therefore filters are not required, and the greater its durability (Albino *et al*, 2011; Alpöz *et al*, 2008; Flury *et al*, 2012; Giorgi *et al*, 2012; Kuguimiya *et al*, 2010). A higher light intensity leads to a higher degree of conversion of the composite resin, resulting in improved physical and mechanical properties (Yaman *et al*, 2011). The energy of the light emitted by the light curing units decreases dramatically when transmitted through the composite, leading to a gradual decrease in the degree of conversion as the distance to the light source increases (Cekig-Nagas, Egilmez and Ergun, 2010; Hedge, Hedge and Malhan, 2008; Voltarelli *et al*, 2009). The decrease in the degree of conversion compromises the physical properties of composites and elution of the monomer increases, which can lead to premature failure of the restoration or even damage to pulpal tissue (Lima *et al*, 2012; Rouhollahi, Mohammadibasir and Talim, 2012). However, a higher degree of polymerization,

caused by a higher light intensity, may result in higher polymerization shrinkage (Voltarelli *et al*, 2009). This justifies also the use of the incremental technique. However, in large cavities, wherein the application of 2 mm thick layers takes more time and is associated with a higher risk of air bubbles and contamination between increments, recent Bulk-fill composites can be useful (El-Safty, Silikas and Watts, 2012; Lima Rouhollahi, Mohammadibasir and Talim, 2012; El-Safty *et al*, 2012).

Objectives:

To evaluate influence of the light curing method on the microhardness and the depth of cure of six bulk-fill composite resins; and to compare two methods used to determine the depth of cure of composite resins according to the followed null hypothesis.

- Light curing method does not influence the microhardness of bulk-fill composite resins.
- Light curing method does not influence the depth of cure of bulk-fill composite resins.
- There are no differences in depth of cure of bulk-fill composite resins determined by ISO or microhardness ratio method.

Materials and methods:

The following resin composites were used: Tetric EvoCeram® Bulk Fill (Ivoclar Vivadent®), x-tra base® (Voco®), x-tra fill (Voco®), Filtek™ Bulk Fill (3M ESPE), SonicFill™ (Kerr™) and SDR™ (Dentsply) (Table1).

Knoop microhardness test and depth of cure based on microhardness ratio

To determine the Knoop microhardness and depth of cure based on the microhardness ratio, 120 specimens were prepared, using a Teflon mould developed by Ivoclar Vivadent (figures 1 and 2). This mould is composed by two pieces to be joined in order to define a parallelepiped shaped cavity with 2 x 2 x 7 mm. One strip of transparent film was placed between the two pieces to guarantee a smooth and regular lateral surface. Specimens were allocated to 24 experimental groups according to the several possible combinations between the 6 bulk-fill composites and the 4 light curing methods used (n=5). The composite resins used were: x-tra base® (Voco®), Filtek™ Bulk Fill (3M ESPE), SDR™ (Dentsply), Tetric EvoCeram® Bulk Fill (Ivoclar Vivadent®), x-tra fill (Voco®) and SonicFill™ (Kerr™), as shown in table 1. The composite resins were applied in a single increment, filling the entire cavity, and light cured through a strip of transparent film, using a light-curing device, bluephase® 20i, Voco® (figure 3), with an output and exposure time according to the correspondent experimental group (600 mW/cm^2 for a period of 20 seconds, 1200 mW/cm^2 for 10 seconds, 600 mW/cm^2 for 40 seconds, 1200 mW/cm^2 for 20 seconds), and exposing the light just on the top of the specimen. Light intensity was controlled with a radiometer bluephase® meter (figure 4). After, light cure, the mould was disassembled to allow access to the lateral surface of the specimen.

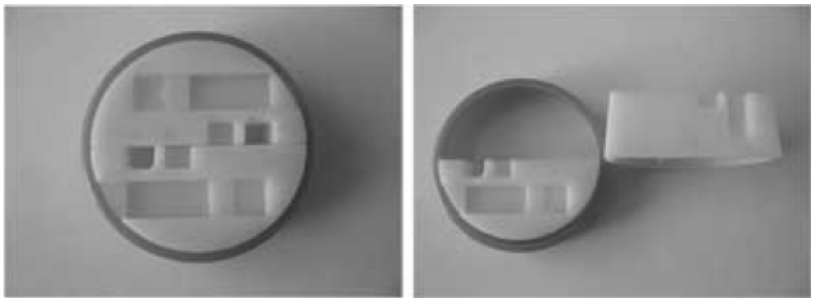


Fig. 1 – Ivoclar Vivadent mould

Composite (shade)	Manufacturer	Composition	Lot	Expiry date
Tetric EvoCeram® Bulk Fill (IVA) Fill	Ivoclar Vivadent®	***Organic matrix*** *(21 wt%):* Bis-GMA, UDMA, Bis-EMA ***Inorganic part*** *(80 wt%; 61 vol%):* Iniciators: camphorquinone, an acyl phosphine oxide and a patented initiator Ivocerin®); Fillers: barium aluminium silicate glass, an „Isofiller“, ytterbium fluoride and spherical mixed oxide	S03808	Jan-2017
Filtek™ Bulk Fill (U) Base	3M ESPE	***Organic matrix:*** Bis-GMA, UDMA, Bis-EMA and Procrylat resins ***Inorganic part*** *64,5 wt%; 42,5 vol%):* Fillers: combination of zirconia/sílica (0,01 - 3,5μ) and ytterbium trifluoride filler (0,1 – 5,0 μ).	N390563	May-2015
x-tra base® (U) Base	Voco®	***Organic matrix:*** *Bis-EMA and MMA* ***Inorganic part****:* *Fillers (75 wt%).*	1251271	Apr-2015
x-tra fil (U) Fill	Voco®	***Organic matrix:*** *No information available* *Inorganic part:* *Fillers (75 wt%).*	1302146	Jan-2015
SDR™ (U) Base	Dentsply	***Organic matrix:*** *SDR*™ patented urethane di-methacrylate resin, di-methacrylate resina and di-fuctional diluent. ***Inorganic part*** *(68 wt%; 45 vol%): Barium and Strontium alumino-fluoro-silicate glasses*	1205022	May-2014
SonicFill™ (A2) Fill	Kerr™	***Organic matrix:*** *No information available* ***Inorganic part:*** *Fillers (83,5 wt%).*	4427310	Mar-2014

Table 1: Materials used in this study and respective manufacturers

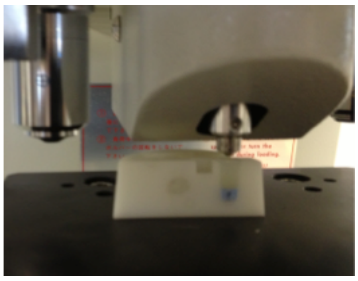


Fig. 2: Top indentation

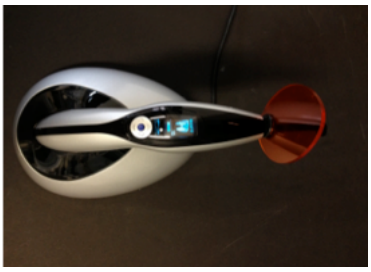


Fig. 3: Bluephase 20i

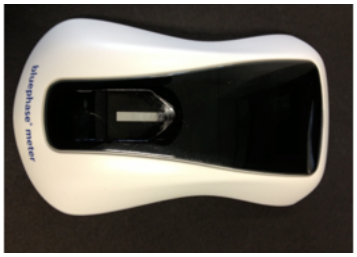


Fig. 4: Radiometer

Three indentations, with a 0.5 mm distance between them, were made on top (figure 2) and on the lateral surface of each specimen, at 1, 2, 3, 4 and 5 mm deep. Indentations were made using Duramin (Struers A/S, DK-2750 Ballerup, Denmark) indenter with a Press Load of 98.12 mN, for 10 seconds (figure 5). The mean value of the three measurements was calculated and used as KHN of each depth for each specimen.

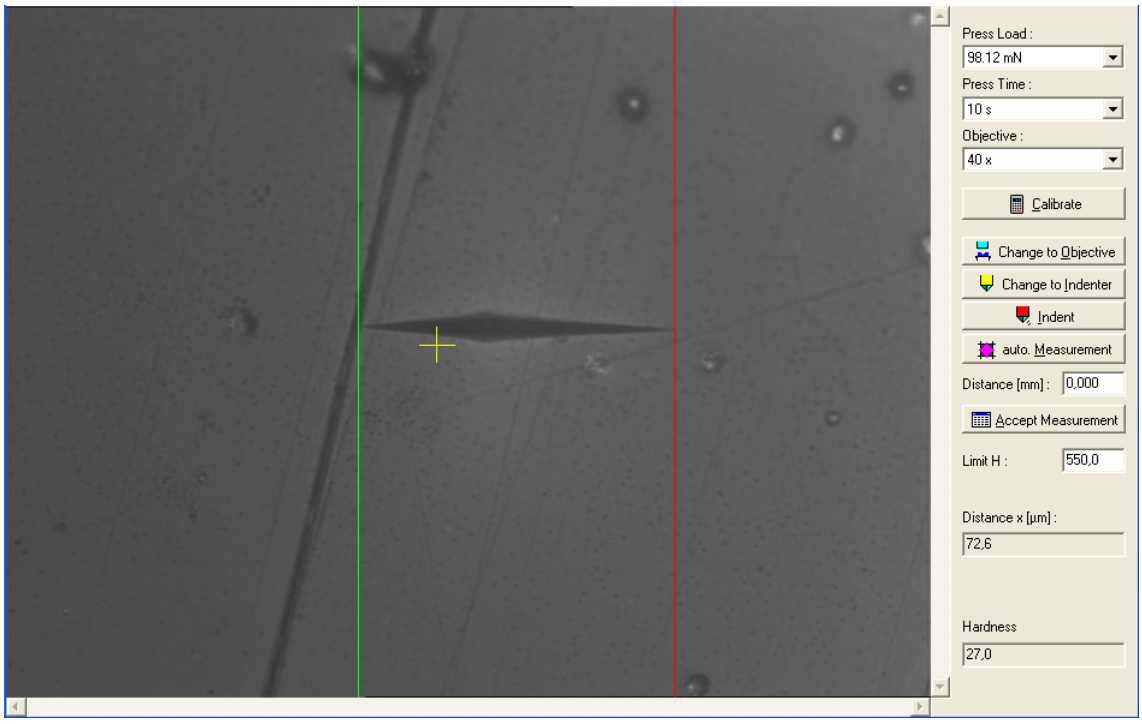


Figure 5: Indentation measurement

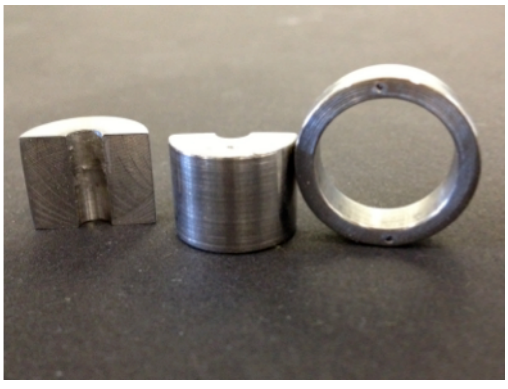
Depth of cure based on ISO 4049

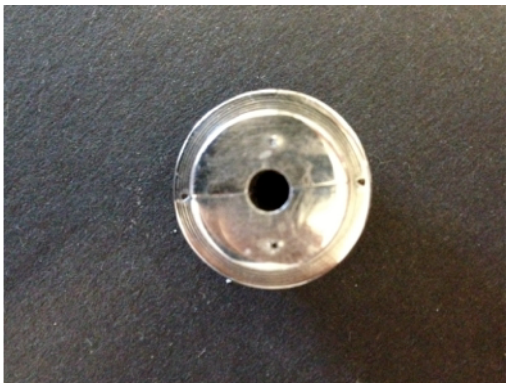
To evaluate the degree of cure by "ISO 4049; Depth of cure", 48 cylindrical specimen with 4 mm in diameter were prepared, using a stainless steel mould (figures 6 and 7). Specimens with 8 mm long were prepared for composite resins with 4 mm claimed depth of cure (all composite resins except SonicFill). Specimens with 10 mm long were prepared with SoniFill.

The resin composites were applied in a single increment.

The specimens were divided into 24 groups (n=2). Four groups were created for each resin composite according to light curing method (600 mW/cm^2 - 20 seconds; 2 1200 mW/cm^2 - 10 seconds; 600 mW/cm^2 - 40 seconds; 1200 mW/cm^2 -20 seconds).

The mould was placed over the acetate strip (transparent film) on a glass microscope slide. The mould was filled with the composite resin according to the manufacturers' instructions, taking care not to let any air bubbles stay. Another acetate strip was placed and then covered with a second glass plate. The glass plates were pressed so as to remove any excess. The mould was placed on the white filter paper; the glass plate was removed from top surface and the output of the light source was placed in contact with the acetate strip. Samples were irradiated as previously described. Immediately after irradiation, the specimens were removed from the mould and the uncured material was scraped with the plastic spatula. The height of the polymerized cylinder was measured with the micrometer to an accuracy of 0.01 mm. The depth of cure was obtained dividing this value by two.





Figures 6 and 7: ISO 4049 stainless steel mould

Statistical analysis

The data concerning the depth of cure were analyzed with descriptive statistics.

The top microhardness data were statistically analyzed using Kruskal-Wallis followed by LSD post-hoc tests by ranks.

A statistical significance of 0,05 was fixed.

Results

The data were inserted into a database and statistically analyzed with computer software – SPSS for Windows, version 21.0 (SPSS Inc. Chicago, IL 60606, EUA).

Top microhardness values ranged from 14,2 (Filtek^{TM} Bulk Fill - 1200mW/cm^2 - 10 seconds) to 51,5 (SonicFill^{TM} -1200mW/cm^2 - 20 seconds), as shown in Table 2.

As neither normality of sample distribution [Kolmogorov-Smirnov test ($p<0.05$)] nor homoscedasticity [Leven test ($p<0.001$)] were observed, Kruskal-Wallis non-parametric tests followed by LSD post-hoc tests by rank were used to analyse microhardness data. Kruskal-Wallis revealed statistically significant differences between composite resins ($p<0.001$). The multiple comparisons according to the LSD method by ranks showed that the composite resins to be used as a base have statistically ($p<0.05$) lower microhardness than those for use as a "fill". Kruskal-Wallis test showed that no statistically significant differences were found between the curing methods ($p=0.244$).

ISO depth of cure ranged between 2.92 mm (Tetric Evoceram Bulk Fill - 600mW/cm^2 - 20s) and 4.97 mm (x-tra base – 600 mW/cm^2 – 40 s). Ratio depth of cure ranged from 4 mm [x-tra fill -1200 mW/cm^2 – 20 s, Filtek Bulk Fill (600 mW/cm^2 – 20 s; 600 mW/cm^2 – 40 s; 1200 mW/cm^2 – 20 s)] to 1 mm [Tetric Evoceram Bulk Fill – (600 mW/cm^2 – 20 s; 1200 mW/cm^2 – 10 s)].

Resin composite	Light Curing Method	Top microhardness: Mean (SD)	DC by ISO	DC by Knoop microhardness
x-tra base® (a)	600mW/cm^2 - 20 s	18.71 (1.83)	4.84	3.00
	1200mW/cm^2 - 10 s	19.87 (3.00)	4.87	3.00
	*600mW/cm^2 - 40 s	26.06 (6.55)	4.85	3.00
	*1200mW/cm^2 - 20 s	26.32 (2.98)	4.76	4.00
Filtek™ Bulk Fill (b)	*600mW/cm^2 - 20 s	16.39 (1.61)	4.13	4.00
	1200mW/cm^2 - 10 s	14.22 (0.94)	4.12	3.00
	600mW/cm^2 - 40 s	15.69 (4.03)	4.79	4.00
	1200mW/cm^2 - 20 s	16.14 (1.01)	4.75	4.00
SDR™ (b)	*600mW/cm^2 - 20 s	17.46 (2.41)	4.03	3.00
	1200mW/cm^2 - 10 s	14.71 (2.61)	4.07	3.00
	600mW/cm^2 - 40 s	15.88 (2.39)	4.53	3.00
	1200mW/cm^2 - 20 s	15.35 (3.13)	4.63	3.00
x-tra fil (c)	*600mW/cm^2 - 20 s	29.82 (4.80)	4.12	2.00
	*1200mW/cm^2 - 10 s	29.73 (4.18)	3.82	2.00
	600mW/cm^2 - 40 s	41.05 (2.72)	4.97	3.00
	1200mW/cm^2 - 20 s	42.97 (1.73)	4.95	3.00
Tetric EvoCeram® Bulk Fill (d)	*600mW/cm^2 - 20 s	33.35 (6.27)	2.92	1.00
	*1200mW/cm^2 - 10 s	28.21 (2.35)	3.13	1.00
	600mW/cm^2 - 40 s	32.41 (2.24)	3.42	2.00
	1200mW/cm^2 - 20 s	31.84 (2.16)	3.61	2.00
SonicFill™ (e)	*600mW/cm^2 - 20 s	41.76 (6.48)	3.01	2.00
	1200mW/cm^2 - 10 s	40.97 (6.12)	2.93	2.00
	600mW/cm^2 - 40 s	48.07 (7.96)	3.15	3.00
	1200mW/cm^2 - 20 s	51.45 (2.62)	3.31	2.00

Table 2: Depth of cure by ISO 4049 and Knoop microhardness tests. The same character means no statistically significant differences in terms of microhardness. *Manufacturer instructions

Discussion:

Physical properties of composite resins depend on their degree of conversion, organic matrix, and filler particles type (Albino *et al*, 2011; Kwon, Ferracane and Lee, 2012). The degree of conversion along the composite bulk can be measured by direct methods, such as infrared spectroscopy (Ferracane, 1985), FT-Raman spectroscopy (Albino *et al*, 2011), or through indirect methods such as ISO 4049 method (Flury *et al*, 2012) or microgardness tests (Ferracane, 1985; DeWald and Ferracane, 1987). Although direct methods are more accurate, microhardness tests are simpler and less expensive and also allow a correlation with the degree of conversion (Ferracane, 1985; DeWald and Ferracane, 1987). The Knoop microhardness test is the most commonly used for polymeric materials, since it minimizes the elastic recovery of these materials (Souza, 1982).

In this study, it was verified that the top microhardness of the composite resins intended for use as base is lower than of the fill composite resins, which are intended to fill the cavity to the surface. The resin composites used as base have lower percentage by weight of filler particles, which give them less strength. Thus, the resin composites classified as base require the use of a conventional composite resin, which has a greater surface hardness.

An adequate degree of conversion to a certain depth from the light irradiated surface can be defined as depth of cure. An inadequate conversion of the monomer to polymer along the restoration may lead to further degradation of the polymeric matrix and failure of the restoration.

According to ISO 4049 method, all tested composites except Tetric EvoCeram Bulk Fill and SonicFill achieved the depth of cure claimed by respective manufacturer. These results are in line with a previous study of Garoushi *et al*, 2013.

The differences between base and fill composite resins could be explained by the different filler loading percentage. In fact, all base composite resins were flowable, presenting a lower amount of filler particles, as expected. Base composite resins showed in general higher depth of cure, which can be related to a more efficient light penetration, due to less amount of filler particles. It is known that the shade of

composite resin also influences depth of cure (Garoushi *et al*, 2013; Leprince et al, 2012; Moore et al, 2008). Trying to eliminate this effect, similar shades, with similar recommended irradiation time were used. It was possible to verify that the composite resins were very similar in shade, except from Tetric EvoCeram Bulk Fill, which was clearly darker and more viscous than the others.

As base bulk-fill composite resins have lower microhardness, it is recommended the use of a harder coating resin composite. Ilie et al, 2013, also describe this necessity since in that study bulk-fill materials showed lower modulus of elasticity and hardness, compared to regular nanohybrid and microhybrid composite resins.

It was found that the method ISO 4049 tends to overestimate the depth of cure of resin composites. This result is in agreement with the study of Flury *et al*, 2012.

Following the manufacturers instructions, measuring depth of cure trough Knoop Microhardness ratio, only Filtek™ Bulk Fill demonstrated the expected depth of cure (4 mm).

Even doubling the light curing time or light intensity, only x-tra base® achieved the pretended depth of cure (4 mm).

Although Tetric EvoCeram Bulk Fill has a new photoinitiator that, according to the manufacturer, would allow greater depth of cure, it does not appear to result.

El-Safty *et al*, 2012, obtained lower hardness values for bulk-fill composite resin, in comparison with the conventional nano-hibrid composite resin. For all resin composites there was a group in which the manufacturer's instructions were followed and other in which, at least, the light curing time or light intensity were doubled. Following the manufacturers instructions, only Filtek™ Bulk Fill demonstrated the expected depth of cure (4 mm).

Contrary to what manufacturers suggest it does not seem possible to polymerize 4 mm deep resin layers, at least following the manufacturer's instructions, concerning to light curing time and light intensity.

The results of this study have clinical interest as they suggest the conditions required to achieve the depth of cure of 4 mm in the composites in which this could achieved.

Further studies are needed in this area, to find the conditions in which the Bulk-fill resin composites can have a degree of conversion adequate to desired depth (4 mm or 5 mm).

Conclusion:

The top microhardness was not affected by the curing method.

Fill composite resins microhardness was higher than base composite's.

Not all composites yielded the depth of cure claimed by manufactures. Following the manufacturers instructions, only Filtek™ Bulk Fill offers a satisfactory degree of cure at 4 mm depth.

The resin composites uses as "fill" showed higher top microhardness than those used as "base".

ISO 4049 recommended method afforded a higher depth of cure than the one obtained by microhardness tests.

References:

Albino L, Rodrigues J, Kawano Y, Cassoni A. Knoop microhardness and FT-Raman evaluation of composite resins: influence of opacity and photoactivation source. Braz Oral Res 2011;25:267-73.

Alpöz AR, Ertugrul F, Cogulu D, Ak AT, Tanoglu M, Kaya E. Effects of Light Curing Method and Exposure Time on Mechanical Properties of Resin Based Dental Materials. Eur J Dent 2008;2:37-42.

Arikawa H, Takahashi H, Minesaki Y, Muraguchi K, Matsuyama T, Kanie T, Ban Seiji. A method for improving the light intensity distribution in dental light-curing units. Dent Mater 2011;30:151-7.

Borges A ,Chasqueira F, Portugal J. Grau de Conversão de Resinas Compostas. Influência do Método de Fotopolimerização. Rev Port Estomatol Med Dent Cir Maxilofac 2009;50:197-203.

Camargo E, Moreschi E, BaseggioW, Cury J, Pascotto R. Composite depth of cure using four polymerization techniques. J Appl Oral Sci. 2009;17:446-50

Carrilho E et al. ^{99m}Tc in the evaluation of microleakage of composite resin restorations with SonicFill^{TM}. An in vitro experimental model. OJST 2012;2:340-7

Cekic-Nagas I, Egilmez F, Ergun G. The Effect of Irradiation Distance on Microhardness of Resin Composites Cured with Different Light Curing Units. Eur J Dent 2010;4:440-6.

Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. Clin Oral Invest 2013;17:227-35.

DeWald JP, Ferracane JL. A comparison of four modes of evaluating depth of cure of light-activated composites. J Dent Res 1987;66:727-30.

El-Safty S, Akhtar R, Silikas N, Watts DC. Nanomechanical properties of dental resin-composites. Dent Mater 2012;28:1292-300.

El-Safty S, Silikas N, Watts DC. Creep deformation of restorative resin-composites intended for bulk-fill placement. Dent Mater 2012;28:928-35.

Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. Dent Mater 1985;1:11-4.

Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Depth of cure of resin composites: Is the ISO 4049 method suitable for bulk fill materials? Dent Mater 2012;28:521-28.

Garoushi S, Säilynoja E, Vallittu PK, Lassila L. Physical properties and depth of cure of a new short fiber reinforced composite. Dent Mater 2013; 21:1-7.

Giorgi MC, Aguiar FH, Soares LE, Liporoni P, Paulillo LA. Does an additional UV LED improve the degree of conversion and Knoop Hardness of light-shade composite resins? Eur J Dent 2012;6:396-401.

Hedge MN, Hegde P, Malhan B. Evaluation of depth of cure and knoop hardness in a dental composite, photo-activated using different methods. J Conserv Dent 2008;11:76–81.

Heintze SD, Zimmerli B. Relevance of In-Vitro Tests of Adhesive and Composite Materials. A Review in 3 Parts. Part 2: Non-standardized tests of composite materials. Schweiz Monatsschr Zahnmed 2011;121:916-23.

Ilie N, Keßler A, Durner J. Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites. J Dent 2013;21:10-16

Katona TR, Winkler MM. Stress analysis of a Bulk-filled Class V light-cured composite restoration. J Dent Res 1994;73:1470-7.

Kuguimiya RN, Alves LB, Seabra FR, Sarmento CF, Santos AS, Machado CT. Influence of light-curing units and restorative materials on the micro hardness of resin cements. Indian J Dent Res 2010;21:49-53.

Kwon Y, Ferracane J, Lee I-B. Effect of layering methods, composite type, and flowable liner on the polymerization shrinkage stress of light cured composites. Dent Mater 2012;28:801-09.

Lima A, Andrade K, Alves L, Soares G, Marchi G, Aguiar F, Peris A, Mitsui F. Influence of light source and extended time of curing on microhardness and degree of conversion of different regions of a nanofilled composite resin. Eur J Dent 2012;6:153-7.

Leprince JG, Leveque P, Nysten B, Gallez B, Devaux J, Leloup G. New insight into the "depth of cure" of dimethacrylate-based dental composites. Dent Mater 2012;28:512-20.

Moore BK, Platt JA, Borges G, Chu T-MG, Katsilieri I. Depth of cure of dental resin composites: ISO 4049 depth and microhardness of types of materials and shades. Oper Dent 2008;33:408-412.

Mousavinasab SM, Meyers I. Comparison of Depth of Cure, Hardness and Heat Generation of LED and High Intensity QTH Light Sources. Eur J Dent 2011;5:299-304.

Papadogiannis D, Tolidis K, Lakes R, Papadogiannis Y. Vidcoelastic properties of low-shrinking composite resin compared to packable composite resins. Dent Mater 2011;30:350-7.

Poggio C, Lombardini M, Gaviati S, Chiesa M. Evaluation of Vickers hardness and depth of cure of six composite resins photo-activated with different polymerization modes J Conserv Dent 2012;15:237–41.

Rouhollahi MR, Mohammadibasir M, Talim Sh. Comparative Depth of Cure Among Two Light-Cured Core Build-Up Composites By Surface Vickers Hardness. J Dent (Tehran) 2012;9:255-61.

Souza SA. Ensaios mecânicos de materiais metálicos. Fundamentos teóricos e práticos, 5th ed. São Paulo: Edgard Blücher; 1982. p. 121-30.

Sudheer V, Manjunath MK. Contemporary curing profiles: Study of efectiveness of cure and polymerization shrinkage of composite resins. J Conserv Dent 2011;14:383-6.

Voltareli FR, dos Santos-Daroz CB, Alves MC, Peris AR, Marchi GM. Effect of different light-curinf devices and aging procedures on composite knoop microhardness.

Braz Oral Res 2009;23:473-9.

Yaman BC, Efes BG, Dörter C, Gömec Y, Erdilek D, Büyükgökçesu S. The effects of halogen and light-emitting diode light curing on the depth of cure and surface microhardness of composite resins. J Conserv Dent 2011;14:136–9.